# **CERTIFICATION**

SDG No:

SUMMARY:

MC46109

Humacao, PR

Laboratory:

Accutest, Massachusetts

Site:

BMS, Building 5 Area, PR

Matrix:

Soil/Groundwater

Soil/groundwater samples (Table 1) were collected on the BMSMC facility – Building 5 Area. The BMSMC facility is located in Humacao, PR. Samples were taken May 25-26, 2016 and were analyzed in Accutest Laboratory of Marlborough, Massachusetts that reported the data under SDG No.: MC46109. Results were validated using the following quality control criteria of the methods employed (MADEP VPH and MAPED EPH, Massachusets Department of Environmental Protection, 2004) and the latest validation guidelines (July, 2015) of the EPA Hazardous Waste Support Section. The analyses performed are shown in Table 1. Individual data review worksheets are enclosed for each target analyte group. The data sample organic data samples summary form shows for analytes results that were qualified.

In summary the results are valid and can be used for decision taking purposes.

Table 1. Samples analyzed and analysis performed

SAMPLE ID	SAMPLE	MATRIX	ANALYSIS PERFORMED
	DESCRIPTION	WIATINA	AMACISIS FEM ONVIED
MC46109-1	RA7-GWS	Groundwater	Volatiles TPHC Ranges
MC46109-1A	RA7-GWS	Groundwater	Extractable TPHC Ranges
MC46109-2	EB05252016	AQ – Equipment Blank	Volatiles TPHC Ranges;
MC46109-2A	EB05252016	AQ – Equipment Blank	Extractable TPHC Ranges
MC46109-3	SB103(2.5-3.5)	Soil	Volatiles TPHC Ranges; Extractable TPHC Ranges
MC46109-4	SB103D(2.5-3.5)	Soil	Volatiles TPHC Ranges; Extractable TPHC Ranges
MC46109-5	SB103D(6-7)	Soil	Volatiles TPHC Ranges; Extractable TPHC Ranges
MC46109-6	MW-235(5-6)	Soil	Volatiles TPHC Ranges; Extractable TPHC Ranges
MC46109-7	SB103-GWD	Groundwater	Volatiles TPHC Ranges
MC46109-7A	SB103-GWD	Groundwater	Extractable TPHC Ranges
MC46109-8	SB103-GWS	Groundwater	Volatiles TPHC Ranges
MC46109-8A	SB103-GWS	Groundwater	Extractable TPHC Ranges

Reviewer Name:

Rafael Infante

Chemist License 1888

Signature:

Date:

June 20, 2016

# Report of Analysis

Page 1 of 1

Client Sample ID: RA7-GWS

Lab Sample ID:

MC46109-1

Matrix:

AQ - Ground Water

MADEP VPH REV 1:1

BMSMC, Building 5 Area, Puerto Rico

05/31/16

**Date Sampled:** 05/25/16

Date Received: 05/27/16

Percent Solids: n/a

Run#1

Method:

Project:

File ID BD73735.D DF 1

Analyzed By AF

**Prep Date** n/a

**Prep Batch** n/a

**Analytical Batch** GBD3652

Purge Volume

Run #1 Run #2

Run #2

5.0 ml

Volatile TPHC Ranges

CAS No.	Compound	Result	RL	MDL	Units	Q
	C5- C8 Aliphatics (Unadi )	ND	50	40	ua/I	

C8 Aliphatics (Unadj.) 50 40 ug/l C9- C12 Aliphatics (Unadj.) ND 50 40 ug/l C9- C10 Aromatics (Unadj.) ND 50 40 ug/l C5- C8 Aliphatics 50 ND 40 ug/l C9- C12 Aliphatics ND 50 ug/l

CAS No. Surrogate Recoveries Run#1 Run# 2 Limits

> 2,3,4-Trifluorotoluene 81% 70-130% 2,3,4-Trifluorotoluene 109% 70-130%



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank



# Report of Analysis

Page 1 of 1

Client Sample ID: RA7-GWS

Lab Sample ID: Matrix:

MC46109-1A

AQ - Ground Water

Method: Project:

MADEP EPH REV 1.1 SW846 3510C

BMSMC, Building 5 Area, Puerto Rico

Date Sampled:

05/25/16

Date Received: 05/27/16

Percent Solids: n/a

File ID DF Prep Date Analyzed By Prep Batch **Analytical Batch** Run #1 DE14447.D 1 06/02/16 TA 05/27/16 OP47664 GDE803

Run #2

Initial Volume

900 ml

Final Volume 2.0 ml

Run#1 Run #2

# **Extractable TPHC Ranges**

CAS No.	Compound	Result	RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.) C9-C18 Aliphatics C19-C36 Aliphatics C11-C22 Aromatics	ND ND ND ND	110 110 110 110	78 78 78 78	ug/l ug/l ug/l ug/l	
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Lim	its	
84-15-1 321-60-8 3386-33-2 580-13-2	o-Terphenyl 2-Fluorobiphenyl 1-Chlorooctadecane 2-Bromonaphthalene	69% 68% 68% 70%		40-1 40-1 40-1 40-1	40% 40%	



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

# Report of Analysis

Page 1 of 1

Client Sample ID: EB05252016 Lab Sample ID: MC46109-2

Matrix: Method: AQ - Equipment Blank

MADEP VPH REV 1/1 BMSMC, Building 5 Area, Puerto Rico Date Sampled: 05/25/16 Date Received: 05/27/16

Percent Solids: n/a

File ID DF Analyzed By **Prep Date** Prep Batch **Analytical Batch** Run#1 BD73732.D 1 05/31/16 AF n/a GBD3652 n/a

Run #2

Project:

Purge Volume

Run#1 Run #2

5.0 ml

**Volatile TPHC Ranges** 

CAS No.	Compound	Result	RL	MDL	Units	Q
	C5- C8 Aliphatics (Unadj.)	ND	50	40	ug/I	
	C9- C12 Aliphatics (Unadj.)	ND	50	40	ug/l	
	C9- C10 Aromatics (Unadj.)	ND	50	40	ug/l	
	C5- C8 Aliphatics	ND	50	40	սբ/l	
	C9- C12 Aliphatics	ND	50	40	ug/l	
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Lim	its	
	2,3,4-Trifluorotoluene	84%		70-1	30%	
	2,3,4-Trifluorotoluene	112%	100	70-1	30%	



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank



# Report of Analysis

Page 1 of 1

Client Sample ID: EB05252016 Lab Sample ID: MC46109-2A

AQ - Equipment Blank

Date Sampled: 05/25/16 Date Received: 05/27/16

Matrix: Method:

MADEP EPH REV 1:1 SW846 3510C BMSMC, Building 5 Area, Puerto Rico

Percent Solids: n/a

Project:

File ID DF Analyzed By **Prep Date** Prep Batch **Analytical Batch** DE14448.D Run#1 1 06/03/16 TA 05/27/16 OP47664 GDE803 Run #2

Initial Volume Final Volume Run#1 970 ml 2.0 ml

Run #2

# **Extractable TPHC Ranges**

CAS No.	Compound	Result	RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.)	ND	100	72	սջ/[	
	C9-C18 Aliphatics	ND	100	72	ug/l	
	C19-C36 Aliphatics	ND	100	72	ug/l	
	C11-C22 Aromatics	ND	100	72	ug/l	
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Lim	its	
84-15-1	o-Terphenyl	83%		40-1	40%	
321-60-8	2-Fluorobiphenyl	68%		40-1	40%	
3386-33-2	I-Chlorooctadecane	86%		40-1	40%	
580-13-2	2-Bromonaphthalene	71%		40-1	40%	



ND = Not detected

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RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank



SB103(2.5-3.5)

Matrix:

MC46109-3 SO - Soil

Method:

Project:

MADEP VPH REV 1.1

BMSMC, Building 5 Area, Puerto Rico

**Date Sampled:** 05/26/16 Date Received:

05/27/16

Percent Solids: 89.6

	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	AB94243.D	1	06/01/16	DF	n/a	n/a	GAB5188
D #5							

Run #2

Run #2

**Initial Weight** Run #1 13.9 g

Final Volume 16.0 ml

Methanol Aliquot

100 ul

**Volatile TPHC Ranges** 

Compound	Result	RL	MDL	Units	Q
C5- C8 Aliphatics (Unadj.)	ND ND	7000 7000	3500 3500	ug/kg	
C9- C10 Aromatics (Unadj.)	ND	7000	3500	ug/kg	
C5- C8 Aliphatics C9- C12 Aliphatics	ND ND	7000 7000	3500 3500		
Surrogate Recoveries	Run# 1	Run# 2	Limi		
2,3,4-Trifluorotoluene 2,3,4-Trifluorotoluene	71% 74%				
	C5- C8 Aliphatics (Unadj.) C9- C12 Aliphatics (Unadj.) C9- C10 Aromatics (Unadj.) C5- C8 Aliphatics C9- C12 Aliphatics Surrogate Recoveries 2,3,4-Trifluorotoluene	C5- C8 Aliphatics (Unadj.) ND C9- C12 Aliphatics (Unadj.) ND C9- C10 Aromatics (Unadj.) ND C5- C8 Aliphatics ND C9- C12 Aliphatics ND Surrogate Recoveries Run# 1 2,3,4-Trifluorotoluene 71%	C5- C8 Aliphatics (Unadj.) ND 7000 C9- C12 Aliphatics (Unadj.) ND 7000 C9- C10 Aromatics (Unadj.) ND 7000 C5- C8 Aliphatics ND 7000 C9- C12 Aliphatics ND 7000 Surrogate Recoveries Run#1 Run# 2 2,3,4-Trifluorotoluene 71%	C5- C8 Aliphatics (Unadj.)       ND       7000       3500         C9- C12 Aliphatics (Unadj.)       ND       7000       3500         C9- C10 Aromatics (Unadj.)       ND       7000       3500         C5- C8 Aliphatics       ND       7000       3500         C9- C12 Aliphatics       ND       7000       3500         Surrogate Recoveries       Run#1       Run#2       Limi         2,3,4-Trifluorotoluene       71%       70-1	C5- C8 Aliphatics (Unadj.) ND 7000 3500 ug/kg C9- C12 Aliphatics (Unadj.) ND 7000 3500 ug/kg C9- C10 Aromatics (Unadj.) ND 7000 3500 ug/kg C5- C8 Aliphatics ND 7000 3500 ug/kg C9- C12 Aliphatics ND 7000 3500 ug/kg C9- C12 Aliphatics ND 7000 3500 ug/kg Surrogate Recoveries Run#1 Run#2 Limits  2,3,4-Trifluorotoluene 71% 70-130%



ND = Not detected

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RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank



# Report of Analysis

Page 1 of 1

Client Sample ID: SB103(2.5-3.5) Lab Sample ID: MC46109-3 Matrix:

SO - Soil

Date Sampled: 05/26/16 Date Received: 05/27/16

Method:

MADEP EPH REV 1.1 SW846 3546

Percent Solids: 89.6

Project:

BMSMC, Building 5 Area, Puerto Rico

File ID DF Analyzed By **Prep Date** Prep Batch **Analytical Batch** Run#1 DE14497.D l 06/08/16 TA 05/30/16 OP47681 **GDE807** 

Run #2

Initial Weight 11.4 g

Final Volume

2.0 ml

Run #1

Run #2

# **Extractable TPHC Ranges**

CAS No.	Compound	Result	RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.) C9-C18 Aliphatics C19-C36 Aliphatics C11-C22 Aromatics	ND ND ND ND	20000 9800 9800 20000	16000 7800 7800 16000	ug/kg ug/kg ug/kg ug/kg	
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limi	its	
84-15-1 321-60-8 580-13-2 3386-33-2	o-Terphenyl 2-Fluorobiphenyl 2-Bromonaphthalene 1-Chlorooctadecane	106% 78% 80% 92%		40-1- 40-1- 40-1-	40% 40%	



ND = Not detected

MDL = Method Detection Limit

J = Indicates an estimated value

RL = Reporting Limit

B = Indicates analyte found in associated method blank

E = Indicates value exceeds calibration range



# Report of Analysis

Page 1 of 1

Client Sample ID: Lab Sample ID:

SB103D(2.5-3.5) MC46109-4

Matrix:

SO - Soil

Method:

MADEP VPH REV 1.1

Project:

BMSMC, Building 5 Area, Puerto Rico

Date Sampled: Date Received:

05/26/16 05/27/16

Percent Solids: 89.7

DF

File ID Run#1 AB94244.D

Analyzed 06/01/16

By DF **Prep Date** n/a

**Prep Batch** n/a

**Analytical Batch** GAB5188

Run #2

Run #2

Initial Weight Run #1 14.6 g

Final Volume 16.0 ml

Methanol Aliquot

100 ul

# Volatile TPHC Ranges

CAS No.	Compound	Result	RL	MDL	Units	Q
	C5- C8 Aliphatics (Unadj.)	ND	6700	3400	ug/kg	
	C9- C12 Aliphatics (Unadj.)	ND	6700	3400	ug/kg	
	C9- C10 Aromatics (Unadj.)	ND	6700	3400	ug/kg	
	C5- C8 Aliphatics	ND	6700	3400	ug/kg	
	C9- C12 Aliphatics	ND	6700	3400	ug/kg	
CAS No.	Surrogate Recoveries	Run# I	Run# 2	Lim	its	
	2,3,4-Trifluorotoluene	68% a		70-1	30%	
	2,3,4-Trifluorotoluene	71%		70-1	30%	

(a) Outside control limits. Refer to Fluorobenzene.



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank



# **Report of Analysis**

Page 1 of I

Client Sample ID: Lab Sample ID:

SB103D(2.5-3.5) MC46109-4

Matrix:

SO - Soil

Method: Project:

MADEP EPH REV 1.1 SW846 3546 BMSMC, Building 5 Area, Puerto Rico

Date Sampled: Date Received:

05/26/16 05/27/16

Percent Solids: 89.7

	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	DE14498.D	1	06/08/16	TA	05/30/16	OP47681	GDE807
Run#2							

Initial Weight 11:1 g

Final Volume 2.0 ml

Run #1 Run #2

# **Extractable TPHC Ranges**

CAS No.	Compound	Result	RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.)	ND	20000	16000	ug/kg	
	C9-C18 Aliphatics	ND	10000	8000	ug/kg	
	C19-C36 Aliphatics	ND	10000	8000	ug/kg	
	C11-C22 Aromatics	ND	20000	16000	ug/kg	
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limi	its	
84-15-1	o-Terphenyl	117%		40-1-	40%	
321-60-8	2-Fluorobiphenyl	86%		40-14	40%	
580-13-2	2-Bromonaphthalene	90%		40-1-	40%	
3386-33-2	1-Chlorooctadecane	94%		40-1	40%	



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank



# **Report of Analysis**

Page 1 of 1

Client Sample ID: SB103(6-7) Lab Sample ID: MC46109-5 Matrix:

SO - Soil

MADEP VPH REV 1.1

Date Sampled: 05/26/16 Date Received: 05/27/16

Method: Project: BMSMC, Building 5 Area, Puerto Rico Percent Solids: 83.6

File ID DF Analyzed By Prep Date Prep Batch **Analytical Batch** Run #1 AB94245.D 1 06/01/16 DF n/a n/a GAB5188 Run #2

	_	un #1	-
N 1			

# **Volatile TPHC Ranges**

CAS No.	Compound	Result	RL	MDL	Units	Q	
	C5- C8 Aliphatics (Unadj.)	10100	8600	4300	ug/kg		
	C9- C12 Aliphatics (Unadj.)	204000	8600	4300	ug/kg		
	C9- C10 Aromatics (Unadj.)	105000	8600	4300	ug/kg		
	C5- C8 Aliphatics	10100	8600	4300	ug/kg		
	C9- C12 Aliphatics	98400	8600	4300	ug/kg		
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Lim	its		
	2,3,4-Trifluorotoluene	72%		70-1	30%		
	2,3,4-Trifluorotoluene	74%		70-1	30%		



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

# **Report of Analysis**

 $\mathbf{B}\mathbf{y}$ 

TA

Page 1 of 1

Client Sample ID: Lab Sample ID:

SB103(6-7) MC46109-5

SO - Soil

Date Sampled: Date Received:

05/26/16 05/27/16

Matrix: Method:

MADEP EPH REV 1.1 SW846 3546

Percent Solids: 83.6

Project:

BMSMC, Building 5 Area, Puerto Rico

Analyzed

06/08/16

Prep Batch OP47681

**Analytical Batch** 

Run#1 Run #2

Prep Date

05/30/16

**GDE807** 

Initial Weight

DE14499.D

File ID

Final Volume

11.0 g

Run#1 Run #2 2.0 ml

DF

# **Extractable TPHC Ranges**

CAS No.	Compound	Result	RL	MDL	Units	Q		
	C11-C22 Aromatics (Unadj.) C9-C18 Aliphatics	71400 336000	22000 11000	17000 8700	ug/kg ug/kg			
	C19-C36 Aliphatics	ND	11000	8700	ug/kg			
	C11-C22 Aromatics	70200	22000	17000	ug/kg			
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limi	its			
84-15-1	o-Terphenyl	105%		40-1-	40%			
321-60-8	2-Fluorobiphenyl	88%		40-140%				
580-13-2	2-Bromonaphthalene	114%		40-1	40%			
3386-33-2	1-Chlorooctadecane	85%		40-140%				





MDL = Method Detection Limit

RL = Reporting Limit E = Indicates value exceeds calibration range J = Indicates an estimated value

B = Indicates analyte found in associated method blank



Method:

# Report of Analysis

Page 1 of 1

Client Sample ID: MW-23S(5-6) Lab Sample ID: MC46109-6 Matrix:

SO - Soil

MADEP VPH REV 1.1

Date Sampled: 05/26/16 Date Received: 05/27/16 Percent Solids: 81.4

Project: BMSMC, Building 5 Area, Puerto Rico

File ID DF Analyzed  $\mathbf{B}\mathbf{y}$ **Prep Date** Prep Batch **Analytical Batch** Run #1 AB94246.D 1 06/01/16 DF n/a **GAB5188** n/a Run #2

	T-242-1 TR7 * 1.4	T: 117.	7.7	
Run#1	initial Weight	Final Volume 16.0 ml	Methanol Aliquot 100 ul	
Run #2	10.5 g	10.0 111	100 til	

# **Volatile TPHC Ranges**

CAS No.	Compound	Result	RL	MDL	Units	Q
	C5- C8 Aliphatics (Unadj.) C9- C12 Aliphatics (Unadj.) C9- C10 Aromatics (Unadj.) C5- C8 Aliphatics C9- C12 Aliphatics	ND 5430 ND ND ND	7200 7200 7200 7200 7200	3600 3600 3600 3600 3600	ug/kg ug/kg ug/kg ug/kg ug/kg	J
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	n#2 Limits		
	2,3,4-Trifluorotoluene 2,3,4-Trifluorotoluene	75% 78%			30% 30%	



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank



# **Report of Analysis**

Page 1 of 1

Client Sample ID: MW-23S(5-6) Lab Sample ID: MC46109-6 Matrix:

SO - Soil

**Date Sampled:** 05/26/16 Date Received: 05/27/16

Method:

MADEP EPH REV 1.1 SW846 3546 BMSMC, Building 5 Area, Puerto Rico Percent Solids: 81.4

Project:

File ID DF Analyzed By Prep Date Prep Batch **Analytical Batch** Run #1 DE14500.D 1 06/08/16 TA 05/30/16 OP47681 **GDE807** 

Run #2

Initial Weight Final Volume Run#1 11.6 g 2.0 ml

Run #2

# **Extractable TPHC Ranges**

CAS No.	No. Compound		RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.) C9-C18 Aliphatics C19-C36 Aliphatics C11-C22 Aromatics	ND ND ND ND	21000 11000 11000 21000	17000 8500 8500 17000	ug/kg ug/kg ug/kg ug/kg	
CAS No.	Surrogate Recoveries	Run#1	Run# 2	ın#2 Limits		
84-15-1 321-60-8 580-13-2 3386-33-2	o-Terphenyl 2-Fluorobiphenyl 2-Bromonaphthalene 1-Chlorooctadecane	112% 79% 79% 93%	40-140% 40-140% 40-140% 40-140%			



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank



# Report of Analysis

Page 1 of 1

Client Sample ID: SB103-GWD Lab Sample ID:

MC46109-7

Matrix:

AQ - Ground Water

Method:

MADEP VPH REV 1.1

Project:

BMSMC, Building 5 Area, Puerto Rico

**Date Sampled:** 05/26/16

Date Received: 05/27/16

Percent Solids: n/a

File ID DF Analyzed By Prep Date **Prep Batch Analytical Batch** Run #1 BD73736.D 1 05/31/16 AF n/a GBD3652 n/a

Run #2

Purge Volume

Run#1 Run #2

5.0 ml

**Volatile TPHC Ranges** 

CAS No.	Compound	Result	RL	MDL	Units	Q
	C5- C8 Aliphatics (Unadj.)	ND	50	40	ug/I	
	C9- C12 Aliphatics (Unadj.)	ND	50	40	ug/l	
	C9- C10 Aromatics (Unadj.)	ND	50	40	ug/l	
	C5- C8 Aliphatics	ND	50	40	ug/l	
	C9- C12 Aliphatics	ND	50	40	ug/l	
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limi	its	
	2,3,4-Trifluorotoluene	83%		70-1	30%	
	2,3,4-Trifluorotoluene	113%		70-1	30%	



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank



# **Report of Analysis**

Page 1 of 1

Client Sample ID: Lab Sample ID:

SB103-GWD MC46109-7A

AQ - Ground Water

Date Sampled: Date Received:

05/26/16 05/27/16

Matrix: Method:

MADEP EPH REV 1.1 SW846 3510C

Project:

BMSMC, Building 5 Area, Puerto Rico

Percent Solids: n/a

Run#1 Run #2

File ID DF DE14449.D 1

Analyzed 06/03/16

By **Prep Date** TA 05/27/16

Prep Batch OP47664

**Analytical Batch GDE803** 

Initial Volume

Final Volume

Run #1 Run #2

900 ml

2.0 ml

# **Extractable TPHC Ranges**

CAS No.	Compound	Result	RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.)	ND	110	78	սբ/[	
	C9-C18 Aliphatics	ND	110	78	ug/l	
	C19-C36 Aliphatics	ND	110	78	ug/l	
	C11-C22 Aromatics	ND	110	78	ug/l	
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Lim	its	
84-15-1	o-Terphenyl	79%		40-1	40%	
321-60-8	2-Fluorobiphenyl	75%		40-1	40%	
3386-33-2	I-Chlorooctadecane	76%		40-1	40%	
580-13-2	2-Bromonaphthalene	77%		40-1	40%	



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

# Report of Analysis

AF

n/a

Page 1 of 1

**Analytical Batch** 

GBD3652

Client Sample ID: Lab Sample ID:

SB103-GWS MC46109-8

Matrix:

AQ - Ground Water

Method:

MADEP VPH REV 1.1

Project:

BMSMC, Building 5 Area, Puerto Rico

05/31/16

Date Sampled:

n/a

Q

05/26/16 05/27/16

Date Received:

Percent Solids: n/a

Prep Batch

File ID DF Analyzed By **Prep Date** 

Run#1 Run #2

BD73738A.D

Purge Volume

Run#1 Run #2 5.0 ml

# **Volatile TPHC Ranges**

CAS No.	Compound	Result	RL	MDL	Units
	C5- C8 Aliphatics (Unadj.)	ND	50	40	ug/l
	C9- C12 Aliphatics (Unadj.)	137	50	40	ug/l
	C9- C10 Aromatics (Unadj.)	83.1	50	40	ug/l
	C5- C8 Aliphatics	ND	50	40	ug/l
	C9- C12 Aliphatics	52.3	50	40	ug/l
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Lim	its
	2,3,4-Trifluorotoluene	79%		70-1	30%
	2,3,4-Trifluorotoluene	104%		70-1	30%



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

# Report of Analysis

By

Page 1 of 1

Client Sample ID: Lab Sample ID:

SB103-GWS MC46109-8A

Matrix:

AQ - Ground Water

DF

1

Date Sampled: Date Received:

05/26/16 05/27/16

Method:

MADEP EPH REV 1.1 SW846 3510C

Percent Solids: n/a

Project:

BMSMC, Building 5 Area, Puerto Rico

Run#1

DE14450.D

File ID

Analyzed 06/03/16 TA **Prep Date** 05/27/16

Prep Batch OP47664

**Analytical Batch** GDE803

Run #2

**Initial Volume** 880 ml

Run#1 Run #2 **Final Volume** 2.0 ml

# **Extractable TPHC Ranges**

CAS No.	Compound	Result	RL	MDL	DL Units		
	C11-C22 Aromatics (Unadj.)	153	110	80	ug/l		
	C9-C18 Aliphatics	189	110	80	ug/l		
	C19-C36 Aliphatics	ND	110	80	ug/l		
	C11-C22 Aromatics	153	110	80	ug/l		
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Lim	its		
84-15-1	o-Terphenyl	83%		40-1	40%		
321-60-8	2-Fluorobiphenyl	73%		40-1	40%		
3386-33-2	1-Chlorooctadecane	86%		40-1	40%		
580-13-2	2-Bromonaphthalene	75%		40-140%			



ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

N = Indicates presumptive evidence of a compound

**ACCUTEST** 

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MC46109: Chain of Custody

Page 1 of 3

### **EXECUTIVE NARRATIVE**

SDG No:

MC46109

Laboratory:

**Accutest, Massachusetts** 

Analysis:

MADEP VPH

Number of Samples:

8

Location:

BMSMC, Building 5 Area

Humacao, PR

**SUMMARY:** 

Eight (8) samples were analyzed for Volatiles TPHC Ranges by method MADEP VPH. Samples were validated following the METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDROCARBONS (VPH) quality control criteria, Massachusetts Department of Environmental Protection, Revision 1.1 (2004). Also the general validation guidelines promulgated by the USEPA Hazardous Wastes Support Section. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

Results are valid and can be used for decision making purposes.

Critical issues:

None

Major:

None

Minor:

None

**Critical findings:** 

None

Major findings:

None

Minor findings:

- 1. Surrogate standard (2,3,4-trifluorotoluene) recovered under the lower laboratory control limits but within generally acceptable control limits. No action taken, professional judgment.
- 2. MS % recovery for the C5-C8 hydrocarbon range under the lower laboratory control limits in sample MC46120-4MS. MS/MSD % recoveries results apply to the unspiked sample. Unspiked samples were from another project. No action taken.

No MS/MSD analyzed in this data package for a soil matrix. No action taken, blank spike/blank spike duplicate % recoveries used to assess accuracy. % recoveries and RPD within laboratory control limits.

COMMENTS:

Results are valid and can be used for decision making purposes.

**Reviewers Name:** 

Rafael Infante

**Chemist License 1888** 

Signature:

June 20, 2016

Date:

# SAMPLE ORGANIC DATA SAMPLE SUMMARY

Sample ID: MC46109-1

Sample location: BMSMC Building 5 Area Sampling date: 5/25/2016

Matrix: Groundwater

# METHOD: MADEP VPH

Ç9 - C12 Aliphatics	Ç5 - C8 Aliphatics	Ç9 - C10 Aromatics (Unadj.)	Ç9 - C12 Aliphatics (Unadj.)	Ç5 - C8 Aliphatics (Unadj.)	Analyte Name
50	50	50	50	50	Result
ug/l	ug/i	ug/I	ug/i	ug/i	Units Dilution F
Ľ	<b>-</b>	F	نسر	دم	tion Factor
,	1	,		,	Lab Flag
C	C	_	C	_	Validation
Yes	Yes	Yes	Yes	Yes	Reportable

Sample ID: MC46109-2

Sample location: BMSMC Building 5 Area

Sampling date: 5/25/2016

Matrix: AQ - Equipment Blank

Ç9 - C12 Aliphatics	Ç5 - C8 Aliphatics	Ç9 - C10 Aromatics (Unadj.)	Ç9 - C12 Aliphatics (Unadj.)	Ç5 - C8 Aliphatics (Unadj.)	Analyte Name
50	50	50	50	50	Result
ug/l 1	ug/l 1	ug/l 1	ug/l 1	ug/l 1	Units Dilution Facto
					7
•	1	•	'	•	Lab Flag Va
<b>C</b>	_	_	<b>C</b>	<b>C</b>	/alidation
Yes	Yes	Yes	Yes	Yes	Reportable

# Sample ID: MC46109-3

Sample location: BMSMC Building 5 Area

Sampling date: 5/26/2016

Matrix: Soil

# METHOD: MADEP VPH

Ç9 - C12 Aliphatics	Ç5 - C8 Aliphatics	Ç9 - C10 Aromatics (Unadj.)	Ç9 - C12 Aliphatics (Unadj.)	Ç5 - C8 Aliphatics (Unadj.)	Analyte Name
7000	7000	7000	7000	7000	Result
ug/kg 1	ug/kg 1	ug/kg 1	ug/kg 1	ug/kg 1	Units Dilution Factor Lab Flag Validation Reportable
	•	•		•	Lab Flag
_	C	C	<b>C</b>	<b>C</b>	Validation
Yes	Yes	Yes	Yes	Yes	Reportable

Sample ID: MC46109-4

Sample location: BMSMC Building 5 Area

Sampling date: 5/26/2016 Matrix: Soil

Analyte Name  Ç5 - C8 Aliphatics (Unadj.)  Ç9 - C12 Aliphatics (Unadj.)  Ç9 - C10 Aromatics (Unadj.)	Result 6700 6700 6700	Units Dilution Factor Lab Flagug/kg 1 - ug/kg 1 - ug/kg 1 - ug/kg 1 - ug/kg 1	Lab Flag	Validation U U U	Reportable Yes Yes Yes Yes Yes
Ç5 - C8 Aliphatics (Unadj.)	6700	ug/kg 1		<b>C</b>	
Ç9 - C12 Aliphatics (Unadj.)	6700	ug/kg 1	•	_	
Ç9 - C10 Aromatics (Unadj.)	6700	ug/kg 1	1	<b>–</b>	
Ç5 - C8 Aliphatics	6700	ug/kg 1	•	C	
Ç9 - C12 Aliphatics	6700	ug/kg 1		C	

# Sample ID: MC46109-5

Sample location: BMSMC Building 5 Area

Sampling date: 5/26/2016

Matrix: Soil

# METHOD: MADEP VPH

Ç9 - C12 Aliphatics	Ç5 - C8 Aliphatics	Ç9 - C10 Aromatics (Unadj.)	Ç9 - C12 Aliphatics (Unadj.)	Ç5 - C8 Aliphatics (Unadj.)	Analyte Name
98400	10100	10500	20400	10100	Result
ug/kg 1	ug/kg 1	ug/kg 1	ug/kg 1	ug/kg 1	Units Dilution Factor Lab Flag Validation Reportable
1	•	•	,	•	Lab Flag
	•		•	•	Validation
Yes	Yes	Yes	Yes	Yes	Reportable

# Sample ID: MC46109-6

Sample location: BMSMC Building 5 Area Sampling date: 5/26/2016

Matrix: Groundwater

Ç9 - C12 Aliphatics	Ç5 - C8 Aliphatics	Ç9 - C10 Aromatics (Unadj.)	Ç9 - C12 Aliphatics (Unadj.)	Ç5 - C8 Aliphatics (Unadj.)	Analyte Name
7200	7200	7200	5430	7200	Result
ug/kg 1	ug/kg 1	ug/kg 1	ug/kg 1	ug/kg 1	Units Dilution Factor Lab Flag Validation
ı	1	•	_	ı	Lab Flag
_	_	_	5	<b>C</b>	Validation
Yes	Yes	Yes	Yes	Yes	Reportable

Sample ID: MC46109-7

Sample location: BMSMC Building 5 Area

Sampling date: 5/26/2016 Matrix: Groundwater

METHOD: MADEP VPH

Ç9 - C12 Aliphatics	Ç5 - C8 Aliphatics	Ç9 - C10 Aromatics (Unadj.)	Ç9 - C12 Aliphatics (Unadj.)	Ç5 - C8 Aliphatics (Unadj.)	Analyte Name
50	50	50	50	50	Result
ug/l 1	ug/l 1	ug/i 1	ug/l 1	ug/l 1	Units Dilution Factor Lab Flag Validation Reportable
•	•	•	•		Lab Flag
<b>C</b>	<b>C</b>	<b>C</b>	<b>C</b>	c	Validation
Yes	Yes	Yes	Yes	Yes	Reportable

Sample ID: MC46109-8

Sample location: BMSMC Building 5 Area Sampling date: 5/26/2016

Matrix: Groundwater

Ç9 - C12 Aliphatics	Ç5 - C8 Aliphatics	Ç9 - C10 Aromatics (Unadj.)	Ç9 - C12 Aliphatics (Unadj.)	Ç5 - C8 Aliphatics (Unadj.)	Analyte Name
52.3	50	83.1	137	50	Result
ug/l 1	ug/l 1	ug/l 1	ug/l 1	ug/l 1	Units Dilution I
•	•	•		•	Factor
			•	•	Lab Flag
	<b>C</b>		C	•	Validation
Yes	Yes	Yes	Yes	Yes	Reportable

# **DATA REVIEW WORKSHEETS**

Type of validation	Full:X	Project Number:_MC4610	)9
	Limited:	Date:05/25-2	6/2016
		Shipping date:05/26	3/2016
		EPA Region: 2	
		<u> </u>	
REVIEW OF	VOLATILE PETROLE	UM HYDROCARBON (VPI	ds) PACKAGE
actions. This document decision and in better according to the data v FOR THE DETER! Massachusetts Depart validation guidelines pr	t will assist the reviewer serving the needs of validation guidance docu WINATION OF VOLUMENT OF Environmental omulgated by the USEP ctions listed on the data	organics were created to deling using professional judgment the data users. The sample ments in the following order of TILE PETROLEUM HYDE Protection, Revision 1.1 (2) A Hazardous Wastes Support in review worksheets are from	It to make more informed results were assessed of precedence METHOD PROCARBONS (VPH), 2004). Also the general Section. The QC criteria
The hardcopied (labora has been reviewed and SVOCs included:	atory name) _Accutest_L d the quality control and	aboratories performance data summariz	_ data package received ed. The data review for
No. of Samples:	8	Sample matrix:Se	
Field blank No.:	-		
Equipment blank 140	<b>-</b>		
Trip blank No.:	MC46109-2		
Field duplicate No.:	MC46109-3/MC46109-		
X Data Complet X Holding Time: N/A GC/MS Tunin N/A Internal Stand X Blanks X Surrogate Rec X Matrix Spike/	s g ard Performance	X Laboratory Control SX Field DuplicatesX CalibrationsX Compound IdentificaX Compound QuantitaX Quantitation Limits	Spikes ations ation
Overall Com (C5_to_C12_Aliphatics;	ments: _Vola :_C9_to_C10_Aromatics	tiles_by_GC_by_Method_MA	DEP_VPH,_REV_1.1
Definition of Qualifiers:			
J- Estimated results U- Compound not R- Rejected data UJ- Estimated nond Reviewer: A M Date: 06/20/2016	detected	*	

		Crite	All ria were not me	criteria were met t and/or see below _	_x
l.	DATA COMPLETNE A. Data Packag				
MISS	ING INFORMATION	DATE LAB. CONTAC	TED <u>[</u>	DATE RECEIVED	
-	2.2.				
B.	Other			Discrepar	ncies:
7. A.72.					
					_

All criteria were metX
Criteria were not met and/or see below

# **HOLDING TIMES**

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of extraction, and subsequently from the time of extraction to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE EXTRACTED	DATE ANALYZED	ACTION
S	amples analyzed	within method re	commended holdi	ng time
			-	
		[		

# Criteria

# Preservation:

Samples analyzed with ambient purge temperature: Samples must be acidified to a pH of 2.0 or less at the time of collection.

Samples analyzed with heated purge temperature: Samples must be treated to a pH of 11.0 or greater at the time of collection.

Methanol preservation of soil/sediment samples is mandatory. Methanol (purge-and-trap grade) must be added to the sample vial before or immediately after sample collection. In lieu of the in-field preservation of samples with methanol, soil samples may be obtained in specially-designed air tight sampling devices, provided that the samples are extruded and preserved in methanol within 48 hours of collection.

# Holding times:

Aqueous samples using ambient or heated purge - analyze within 14 days. Soil/sediment samples - analysis within 28 days.

Cooler temperature	: (Criteria:	4 <u>+</u> 2 ℃):_	5.9°C	
--------------------	--------------	-------------------	-------	--

Actions: Qualify positive results/nondetects as follows:

If holding times are exceeded, estimate positive results (J) and nondetects (UJ).

If holding times are grossly exceeded, use professional judgment to qualify data. The data reviewer may choose to estimate positive results (J) and rejects nondetects (R).

If samples were not at the proper temperature (> 10°C) or improperly preserved, use professional judgment to qualify the results.

		eria were metX
	Criteria were not met ar	nd/or see below
CALIBRATIONS VERIFICATION		
Compliance requirements for satisfactory in that the instrument is capable of producing a		
Date of initial calibration:01/12/16	·	02/19/16
Dates of initial calibration verification:	:01/12/16_	02/19/16
Instrument ID numbers:GC	:AB	GCBD
Matrix/Level:AC	QUEOUS/MEDIUM	
DATE LARFILE ANALYTE	CRITERIA OUT	SAMPLES

ID#	MAKETTE	RFs, %RSD, %D, r	AFFECTED
icv5058-50	rt5.5-7	22.6	None
			'
	·		
	ID#	ID#	ID# RFs, %RSD, %D, r

Note: Initial and initial calibration verification meet method specific requirements except for the cases described in this document. No action taken, professional judgment.

# Criteria- ICAL

- Five point calibration curve.
- The percent relative standard deviation (%RSD) of the calibration factor must be equal to or less than 25% over the working range for the analyte of interest. When this condition is met, linearity through the origin may be assumed, and the average calibration factor is used in lieu of a calibration curve.
- A collective calibration factor must also be established for each hydrocarbon range of
  interest. Calculate the collective CFs for C5-C8 Aliphatic Hydrocarbons and C9-C12
  Aliphatic Hydrocarbons using the FID chromatogram. Calculate the collective CF for
  the C9-C10 Aromatic Hydrocarbons using the PID chromatogram. Tabulate the
  summation of the peak areas of all components in that fraction against the total
  concentration injected. The %RSD of the calibration factor must be equal to or less
  than 25% over the working range for the hydrocarbon range of interest.

### DATA REVIEW WORKSHEETS

# Criteria- CCAL

- At a minimum, the working calibration factor must be verified on each working day, after every 20 samples, and at the end of the analytical sequence by the injection of a mid-level continuing calibration standard to verify instrument performance and linearity.
- If the percent difference (%D) for any analyte varies from the predicted response by more than ±25%, a new five-point calibration must be performed for that analyte. Greater percent differences are permissible for n-nonane. If the %D for n-nonane is greater than 30, note the nonconformance in the case narrative. It should be noted that the %Ds are calculated when CFs are used for the initial calibration and percent drifts are calculated when calibration curves using linear regression are used for the initial calibration.

### Actions:

If %RSD > 25% for target compounds or a correlation coefficient < 0.99, estimate positive results (J) and use professional judgment to qualify nondetects. If % D > 25% (> 30 for nonane), estimate positive results (J) and nondetects (UJ).

# CALIBRATIONS VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of init	02/19/16						
Dates of co	Dates of continuing calibration verification:06/01/16						
Dates of fir	nal calibration v	erification:	06/01/16	05/31/16			
Instrument	GCBD_						
Matrix/Leve	Matrix/Level:AQUEOUS/MEDIUM						
DATE	LAB FILE	ANALYTE	CRITERIA OUT	SAMPLES			
	! <b>ID#</b> `		RFs %RSD %D r	AFFECTED			

	DATE	LAB FILE ID# \	ANALYTE	CRITERIA OUT RFs, %RSD, %D, r	SAMPLES AFFECTED
		1011		1(1 3, 701(0), 700, 1	ALLEGIED
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				]	

Note: Continuing and final calibration verification meet method and guidance document specific requirements. Ending calibration verification included in data package.

A separate worksheet should be filled for each initial curve.

				Criteria were r	All criteria were met not met and/or see below	
VA.	BLANK A	ANALYSIS RE	SULTS (Se	ctions 1 & 2)		
of corassoci with a determ proble be ru	ntaminatio iated with any blanks nine whet em is an is	n problems. the samples, s exist, all da her or not the olated occurre amples suspe	The criterial including transcript tales associated received in the criterian criterian increased in the criterian c	for evaluation of the following in the case of the case. As the case of the ca	ne the existence and man of blanks apply only to d laboratory blanks. If promust be carefully evalu- not the data for the case, of A Laboratory Method Blar ninated to determine if	blanks oblems ated to or if the old must
List the		ination in the	blanks bel	ow. High and low	levels blanks must be	treated
Labor	atory blani	ks				
DATE ANAL	: YZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS	
ME	THOD BL	ANKS MEET 1	THE METHO	DD SPECIFIC CRI	TERIA	
Field/	Trip/ <u>Equip</u>	ment				
each					should continually accortively, during sampling, s	
DATE	: .YZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS	
_NO_ _BLAI	TARGET_ NKS_ASS	ANALYTES_I	DETECTED TH_THIS_D	_IN_THE_EQUIPN ATA_PACKAGE	MENT_BLANK.NO_TRIP/	FIELD
					3.7 NE - 9	

### DATA REVIEW WORKSHEETS

All criteria were met _	_X
Criteria were not met and/or see below _	

# V B. BLANK ANALYSIS RESULTS (Section 3)

# Blank Actions

The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. Peaks must not be detected above the Reporting Limit within the retention time window of any analyte of interest. The hydrocarbon ranges must not be detected at a concentration greater than 10% of the most stringent MCP cleanup standard. Specific actions area as follows:

If the concentration is < sample quantitation limit (SQL) and < AL, report the compound as not detected (U) at the SQL.

If the concentration is  $\geq$  SQL but < AL, report the compound as not detected (U) at the reported concentration.

If the concentration is > AL, report the concentration unqualified.

All criteria were met	
Criteria were not met and/or see below	Х

# SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery. Matrix: solid/aqueous

SAMPLE ID

SURROGATE COMPOUND

**ACTION** 

2.3.4-Trifluorotoluene

\_SURROGATE\_STANDARD\_RECOVERIES\_WITHIN\_LABORATORY\_CONTROL\_LIMITS \_EXCEPT\_FOR\_THE\_CASES\_DESCRIBED\_IN\_THIS\_DOCUMENT\_\_

Lab

Lab

Sample ID MC46109-4

File ID S1 a AB94244.D

68\* c 71

**S1** b

Surrogate Compounds Recovery Limits

S1 = 2,3,4-Trifluorotoluene

70-130%

- (a) Recovery from GC signal #2
- (b) Recovery from GC signal #1
- (c) Outside control limits. Refer to Fluorobenzene.

**Note:** No action taken, professional judgment.

It is recommended that surrogate standard recoveries be monitored and documented on a continuing basis. At a minimum, when surrogate recovery from a sample, blank, or QC sample is less than 70% or more than 130%, check calculations to locate possible errors, check the fortifying standard solution for degradation, and check changes in instrument performance.

If the cause cannot be determined, reanalyze the sample unless one of the following exceptions applies:

- Obvious interference is present on the chromatogram (e.g., unresolved (1) complex mixture);
- Percent moisture of associated soil/sediment sample is >25% and surrogate (2)recovery is >10%; or
- The surrogate exhibits high recovery and associated target analytes or (3)hydrocarbon ranges are not detected in sample.

# **DATA REVIEW WORKSHEETS**

If a sample with a surrogate recovery outside of the acceptable range is not reanalyzed based on any of these aforementioned exceptions, this information must be noted on the data report form and discussed in the Executive Report. Analysis of the sample on dilution may diminish matrix-related surrogate recovery problems. This approach can be used as long as the reporting limits to evaluate applicable MCP standards can still be achieved with the dilution. If not, reanalysis without dilution must be performed.

All criteria were met	
Criteria were not met and/or see below_	Χ

# VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples.

At the request of the data user, and in consideration of sample matrices and data quality objectives, matrix spikes and matrix duplicates may be analyzed with every batch of 20 samples or less per matrix.

- Matrix duplicate Matrix duplicates are prepared by analyzing one sample in duplicate. The purpose of the matrix duplicates is to determine the homogeneity of the sample matrix as well as analytical precision. The RPD of detected results in the matrix duplicate samples must not exceed 50 when the results are greater than 5x the reporting limit.
- The desired spiking level is 50% of the highest calibration standard. However, the total concentration in the MS (including the MS and native concentration in the unspiked sample) should not exceed 75% of the highest calibration standard in order for a proper evaluation to be performed. The purpose of the matrix spike is to determine whether the sample matrix contributes bias to the analytical results. The corrected concentrations of each analyte within the matrix spiking solution must be within 70 130% of the true value. Lower recoveries of n-nonane are permissible (if included in the calibration of the C9-C12 aliphatic range), but must be noted in the narrative if <30%.</p>

Sample ID:MC46120-4 Matrix/					/Level:	_Groun	dwater/	low		
List the %Rs, RPD of the compounds which do not meet the QC criteria.										
The QC reported here applies to the following samples: MC46109-1, MC46109-2, MC46109-7, MC46109-8					Metho	d: MADE	P VPH	REV 1.1		
Compound	MC466 ug/kg	120-4 Q	Spike ug/l	MS ug/l	MS %	Spike ug/l	MSD ug/l	MSD %	RPD	Limits Rec/RPD

99.4

150

MS/MSD Recoveries and Precision Criteria

ND

C5- C8 Aliphatics

(Unadi.)

**Note:** MS/MSD % recoveries results apply to the unspiked sample. Unspiked samples were from another project. No action taken.

No MS/MSD analyzed in this data package for a soil matrix. No action taken, blank spike/blank spike duplicate % recoveries used to assess accuracy. % recoveries and RPD within laboratory control limits.

66\* a 150

106

71

6

70-130/25

<sup>(</sup>a) Outside control limits due to possible matrix interference.

<sup>\* =</sup> Outside of Control Limits.

			Criteria w		ia were metX I/or see below
No action is taken of informed professions conjunction with other in those instances who sample spiked, the operation of the analysis of one	al judgment, the QC criteria and nere it can be de ualification shouthe MS/MSD res	ne data d deterr etermine uld be li sults tha	reviewer in the need that the remainded to this the laborar	may use the ed for some qua esults of the MS s sample alone. tory is having a	MS/MSD results in alification of the data. S/MSD affect only the However, it may be a systematic problem
2. MS/MSD – Ur	nspiked Compou	ınds			
List the concentratio compounds in the un					
COMPOUND	CONCENTRATE SAMPLE	TION MS	MSD	%RPD	ACTION
1900		_		- 150-0	
					<del></del>
Criteria: None specific	ed, use %RSD <	≤ 50 as <sub>l</sub>	professional	judgment,	
Actions:					
If the % RSD > 50, qu If the % RSD is not couse professional judg	alculable (NC) d	lue to no	ondetect val		

A separate worksheet should be used for each MS/MSD pair.

12	All criteria were met _	_X
Criteria were not	met and/or see below	

# VIII. LABORATORY CONTROL SAMPLE (LCS/LCSD) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

LCS Recoveries Criteria

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT	ACTION	
LCS_RE	ECOVERY_WITHIN_L	ABORATORY	_CONTROL_LIM	TS	
		01 67 1577		21 ESTRUCTO	

# Criteria:

- Refer to QAPP for specific criteria.
- \* The spike recovery must be between 70% and 130%. Lower recoveries of nnonane are permissible (if included in the calibration of the C9-C12 aliphatic range). If the recovery of n-nonane is <30%, note the nonconformance in the executive narrative.

# Actions:

Actions on LCS recovery should be based on both the number of compounds that are outside the %R criteria and the magnitude of the excedance of the criteria.

If the %R of the analyte is > UL, qualify all positive results (j) for the affected analyte in the associated samples and accept nondetects.

If the %R of the analyte is < LL, qualify all positive results (j) and reject (R) nondetects for the affected analyte in the associated samples.

If more than half the compounds in the LCS are not within the required recovery criteria, qualify all positive results as (J) and reject nondetects (R) for all target analyte(s) in the associated samples.

# 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix (1 per 20 samples per matrix)? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected. Discuss the actions below:

			Criteria were not n		were metX r see below	
IX. FIELD/LAE	BORATOR	Y DUPLICATE PR	ECISION			
Sample IDs:	MC4610	9-3/.C46109-4		Matrix:	Soil	
precision. These a have more varia performance. It is	analyses mability that also expe	neasure both field n laboratory du cted that soil dupli	ken and analyzed and lab precision; plicates which mocate results will have the collecting identicate.	therefore easures /e a grea	, the results ma only laborato ter variance tha	ay ory ar
COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION	
Field duplicate an			e. RPD within guida centration > 5 x SQ		ment criteria	
Criteria:						
RPD <u>+</u> 30% for aq	ueous san	nples, RPD <u>+</u> 50 %	ct-specific information for solid samples it RPD criteria is doub	results a	ire ≥ SQL.	
SQL = soil quantita	ation limit					
Actions:						
If both the sample (NC). No action is		uplicate results are	e nondetects (ND),	the RPD	is not calculab	ılε
Qualify as estimated exceeded the above		ve results (J) an	d nondetects (UJ)	for the	compound th	a
If one sample resu	ılt is not de	tected and the oth	er is $\geq$ 5x the SQL of	qualify (J/	UJ).	
		ple and duplicate lification is appropr	are significantly d	lifferent,	use profession	ıa

If one sample value is not detected and the other is < 5x the SQL, use professional judgment to determine if qualification is appropriate.

All criteria were metX_	
Criteria were not met and/or see below	

### XI. COMPOUND IDENTIFICATION

The compound identification evaluation is to verify that the laboratory correctly identified target analytes as well as tentatively identified compounds (TICs).

- 1. Verify that the target analytes were within the retention time windows.
  - Retention time windows must be re-established for each Target VPH Analyte each time a new GC column is installed, and must be verified and/or adjusted on a daily basis.
  - o Coelution of the m- and p- xylene isomers is permissible.
  - o All surrogates must be adequately resolved from individual Target Analytes included in the VPH Component Standard.
  - For the purposes of this method, adequate resolution is assumed to be achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks.
  - The n-pentane (C5) and MTBE peaks must be adequately resolved from any solvent front that may be present on the FID and PID chromatograms, respectively.

Note: Target analytes were within the retention time window.

2. If target analytes and/or TICs were not correctly identified, request that the laboratory resubmit the corrected data.

		All criteria were metX
	Criteria were	e not met and/or see below
XII. QUANTITATION	I LIMITS AND SAMPLE RESULTS	
The sample quantitation	evaluation is to verify laboratory qu	uantitation results.
1. In the space belo	ow, please show a minimum of one	sample calculation:
Blank Spike	VPH (C9 - C12 Aliphatics)	$RF = 6.167 \times 10^5$
FID		
[] = (31195386)/(6.167	x 10⁵)	
[] = 50.58 ppb Ok		
Blank Spike	VPH (C9 – C10 Aromatics)	$RF = 4.917 \times 10^5$
PID		
[] = (20785602)/(4.917	× 10⁵)	
[] = 42.27 ppb Ok		
2. If requested, ver (MDLs).	ify that the results were above the	laboratory method detection limit
	rmed, were the SQLs elevated ac ples and dilution factor in the table	
SAMPLE ID	DILUTION FACTOR	REASON FOR DILUTION
	,	
	rmed and the results were above the compounds. List the affected same	

#### **EXECUTIVE NARRATIVE**

SDG No:

MC46109

Laboratory:

**Accutest, Massachusetts** 

Analysis:

**MADEP EPH** 

**Number of Samples:** 

Location:

BMSMC, Building 5 Area

Humacao, PR

SUMMARY:

Eight (8) samples were analyzed for Extractable TPHC Ranges by method MADEP EPH. Samples were validated following the METHOD FOR THE DETERMINATION OF EXTRACTABLE PETROLEUM HYDROCARBONS (EPH) quality control criteria, Massachusetts Department of Environmental Protection, Revision 1.1 (2004). Also the general validation guidelines promulgated by the USEPA Hazardous Wastes Support Section. The QC criteria and data validation actions listed on the data review worksheets

are from the primary guidance document, unless otherwise noted.

Results are valid and can be used for decision making purposes.

Critical issues:

None

Major:

None

Minor:

None

**Critical findings:** 

None

Major findings:

None

Minor findings:

1. No MS/MSD analyzed for aqueous matrix in this data package. No action taken, blank

spike/blank spike duplicate used to assess accuracy. % recoveries and RPD within

laboratory control limits.

**COMMENTS:** 

Results are valid and can be used for decision making purposes.

Reviewers Name:

Rafael Infante

Chemist License 1888

Signature:

June 20, 2016

Date:

### SAMPLE ORGANIC DATA SAMPLE SUMMARY

. . . .

Sample ID: MC46109-1A

Sample location: BMSMC Building 5 Area

Sampling date: 5/25/2016

Matrix: Soil

## METHOD: MADEP EPH

Analyte Name	Result	Units D	Dilution Factor	Lab Flag	Validation	Reportable
Ç11 - C22 Aromatics (Unadj.)	110	ug/i	1	-	υ	Yes
Ç9 - C18 Aliphatics	110	ug/l	1	-	U	Yes
Ç19 - C36 Aliphatics	110	ug/l	1	-	U	Yes
Ç11 - C22 Aromatics	110	ug/l	1	-	U	Yes

Sample ID: MC46109-2A

Sample location: BMSMC Building 5 Area

Sampling date: 5/25/2016

Matrix: AQ - Equipment Blank

### METHOD: MADEP EPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Ç11 - C22 Aromatics (Unadj.)	100	ug/i	1	-	IJ	Yes
Ç9 - C18 Aliphatics	100	ug/l	1	-	U	Yes
Ç19 - C36 Aliphatics	100	ug/l	1	-	υ	Yes
Ç11 - C22 Aromatics	100	ug/l	1		U	Yes

Sample ID: MC46109-3

Sample location: BMSMC Building 5 Area

Sampling date: 5/26/2016

Matrix: Soil

# METHOD: MADEP EPH

Analyte Name	Result	Units D	ilution Factor	Lab Flag	Validation	Reportable
Ç11 - C22 Aromatics (Unadj.)	20000	ug/kg	1	•	U	Yes
Ç9 - C18 Aliphatics	9800	ug/kg	1		U	Yes
Ç19 - C36 Aliphatics	9800	ug/kg	1	-	IJ	Yes
Ç11 - C22 Aromatics	20000	ug/kg	1	-	U	Yes

Sample ID: MC46109-4

Sample location: BMSMC Building 5 Area

Sampling date: 5/26/2016

Matrix: Soil

### METHOD: MADEP EPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Ç11 - C22 Aromatics (Unadj.)	20000	ug/kg	1	-	υ	Yes
Ç9 - C18 Aliphatics	10000	ug/kg	1	-	U	Yes
Ç19 - C36 Aliphatics	10000	ug/kg	1	-	U	Yes
Ç11 - C22 Aromatics	20000	ug/kg	1	-	U	Yes

Sample ID: MC46109-5

Sample location: BMSMC Building 5 Area

Sampling date: 5/26/2016

Matrix: Soil

### METHOD: MADEP EPH

Analyte Name	Result	Units D	ilution Factor	Lab Flag	Validation	Reportable
Ç11 - C22 Aromatics (Unadj.)	71400	ug/kg	1	-	(.5)	Yes
Ç9 - C18 Aliphatics	33600	ug/kg	1	-	-	Yes
Ç19 - C36 Aliphatics	11000	ug/kg	1	-	υ	Yes
Ç11 - C22 Aromatics	70200	ug/kg	1	_	-	Yes

Sample ID: MC46109-6

Sample location: BMSMC Building 5 Area

Sampling date: 5/26/2016

Matrix: Soil

# METHOD: MADEP EPH

Analyte Name	Result	Units	<b>Dilution Factor</b>	Lab Flag	Validation	Reportable
Ç11 - C22 Aromatics (Unadj.)	21000	ug/kg	1	-	υ	Yes
Ç9 - C18 Aliphatics	11000	ug/kg	1	-	U	Yes
Ç19 - C36 Aliphatics	11000	ug/kg	1		U	Yes
Ç11 - C22 Aromatics	21000	ug/kg	1		U	Yes

i ype of validation	Full:X Limited:	Project Number:_MC46109 Date:05/25-26/2016
	Littled.	Shipping date:05/26/2016
		EPA Region: 2
REVIEW OF E	XTRACTABLE PETRO	DLEUM HYDROCARBON (EPHs) PACKAGE
validation actions. T more informed decis were assessed according to the precedence METH HYDROCARBONS (2004). Also the ge Support Section. The	his document will assist sion and in better serving ording to the data validation FOR THE DETE (VPH), Massachusetts Deneral validation guideling	latile organics were created to delineate required the reviewer in using professional judgment to make a the needs of the data users. The sample results ation guidance documents in the following order of ERMINATION OF EXTRACTABLE PETROLEUM repartment of Environmental Protection, Revision 1.1 respromulgated by the USEPA Hazardous Wastes alidation actions listed on the data review worksheets less otherwise noted.
The hardcopied (la received has been re review for SVOCs in	eviewed and the quality	test_Laboratories data package control and performance data summarized. The data
No. of Samples:	8	Sample matrix:Soil/Groundwater
Trip blank No.:	MC46109-2_	
Field duplicate No.:	MC46109-3/MC461	09-4
X Data Comp X Holding Tir N/A GC/MS Tur N/A internal Sta X Blanks	pleteness mes	X Laboratory Control SpikesX Field DuplicatesX CalibrationsX Compound IdentificationsX Compound Quantitation
Overall _Extractable_Petrole (C9_to_C36_Aliphati	eum_Hydrocarbons_by_0 ics;_C11_to_C22_(Arom	Comments: GC_by_Method_MADEP_EPH,_REV_1.1 atics)
Definition of Qualifier	rs:	
J- Estimated re U- Compound n R- Rejected dat UJ- Estimated n Reviewer:  Date: 06/20/2016	not detected	
_		

	Criteria were not	All criteria were metx t met and/or see below
I. DATA COMPLETNE A. Data Packag		
MISSING INFORMATION	DATE LAB. CONTACTED	DATE RECEIVED
B. Other		Discrepancies:
	X	

All criteria were met	_X
Criteria were not met and/or see below	

### **HOLDING TIMES**

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of extraction, and subsequently from the time of extraction to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE EXTRACTED	DATE ANALYZED	ACTION
Samples	extracted and ar	nalyzed within me	thod recommend	ed holding time

### Criteria

#### Preservation:

Aqueous samples must be acidified to a pH of 2.0 or less at the time of collection.

Soil samples must be cooled at 4 ± 2 °C immediately after collection.

### Holding times:

Samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction.

Cooler temperature (Criteria: 4 + 2 °C): 5.9°C

Actions: Qualify positive results/nondetects as follows:

If holding times are exceeded, estimate positive results (J) and nondetects (UJ). If holding times are grossly exceeded, use professional judgment to qualify data. The data reviewer may choose to estimate positive results (J) and rejects nondetects (R). If samples were not at the proper temperature (> 10°C) or improperly preserved, use professional judgment to qualify the results.

		Crite	All criteria eria were not met and/o	a were metX or see below		
CALIBRAT	IONS VERIFIC	ATION				
Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.						
Date of initial calibration:02/04/16						
		Dates of i	initial calibration verific	ation:_02/04/13		
Instrument ID numbers:GCDE						
		Matrix/Le	vel:AQU	EOUS/MEDIUM		
DATE	LAB FILE ID#	ANALYTE	CRITERIA OUT RFs, %RSD, %D, r	SAMPLES AFFECTED		
	nitial and conti	nuing calibration med	et method specific requ	uirements		

#### Criteria- ICAL

- Five point calibration curve.
- The percent relative standard deviation (%RSD) of the calibration factor must be equal to or less than 25% over the working range for the analyte of interest.
   When this condition is met, linearity through the origin may be assumed, and the average calibration factor is used in lieu of a calibration curve.
- A collective calibration factor must also be established for each hydrocarbon range of interest. Calculate the collective CFs for C9-C18 Aliphatic Hydrocarbons, C19-C36 Aliphatic Hydrocarbons, and C11-C22 Aromatic Hydrocarbons using the FID chromatogram. Tabulate the summation of the peak areas of all components in that fraction against the total concentration injected. The %RSD of the calibration factor must be equal to or less than 25% over the working range for the hydrocarbon range of interest.
  - The area for the surrogates must be subtracted from the area summation of the range in which they elute.
  - The areas associated with naphthalene and 2-methylnaphthalene in the aliphatic range standard must be subtracted from the uncorrected collective C9-C18 Aliphatic Hydrocarbon range area prior to calculating the CF.

### Criteria- CCAL

 At a minimum, the working calibration factor must be verified on each working day, after every 20 samples or every 24 hours (whichever is more frequent), and

- at the end of the analytical sequence by the injection of a mid-level continuing calibration standard to verify instrument performance and linearity.
- If the percent difference (%D) for any analyte varies from the predicted response by more than ±25%, a new five-point calibration must be performed for that analyte. Greater percent differences are permissible for n-nonane. If the %D for n-nonane is greater than 30, note the nonconformance in the case narrative. It should be noted that the %Ds are calculated when CFs are used for the initial calibration and percent drifts are calculated when calibration curves using linear regression are used for the initial calibration.

#### Actions:

If %RSD > 25% for target compounds or a correlation coefficient < 0.99, estimate positive results (J) and use professional judgment to qualify nondetects.

If % D > 25% (> 30 for nonane), estimate positive results (J) and nondetects (UJ).

#### CALIBRATIONS VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:02/04/16	
Dates of continuing calibration verification:_06/0	2/16;_06/08/16
Dates of final calibration verification:06/0	3/16;_06/08/16
Instrument ID numbers:GCDE	
Matrix/Level:SOIL/AQUEOUS/MEDIUM	

DATE	LAB FILE ID#	ANALYTE	CRITERIA OUT RFs, %RSD, %D, r	SAMPLES AFFECTED
Initial and		ibration meets meth verification included	nod specific requirement d in data package.	s. Final calibration

A separate worksheet should be filled for each initial curve

				All criteria were metX met and/or see below
VA. BLANK	( ANALYSIS R	ESULTS (Se	ctions 1 & 2)	
magnitude of oblanks associate problems with evaluated to do case, or if the	contamination ated with the s any blanks of letermine whe problem is ar must be run	problems. The samples, incluexist, all data ther or not the isolated occurrence after sample	e criteria for evaluding trip, equipma associated with ere is an inherenturrence not affects suspected of l	etermine the existence and uation of blanks apply only to nent, and laboratory blanks. It is the case must be carefully to variability in the data for the cting other data. A Laboratory being highly contaminated to
List the contar separately.	mination in the	e blanks belov	w. High and low i	levels blanks must be treated
Laboratory bla	nks			
DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
METHOD B	LANKS MEET	THE METHO		ITERIA
Field/Trip/Equi	pment			
DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
				MENT_BLANKNO_TRIP/_ S_DATA_PACKAGE
		<del>.</del>		

All criteria were metX_	_
Criteria were not met and/or see below	

# V B. BLANK ANALYSIS RESULTS (Section 3)

#### **Blank Actions**

The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. Peaks must not be detected above the Reporting Limit within the retention time window of any analyte of interest. The hydrocarbon ranges must not be detected at a concentration greater than 10% of the most stringent MCP cleanup standard. Specific actions area as follows:

If the concentration is < sample quantitation limit (SQL) and < AL, report the compound as not detected (U) at the SQL.

If the concentration is  $\geq$  SQL but < AL, report the compound as not detected (U) at the reported concentration.

If the concentration is > AL, report the concentration unqualified.

CAMPLEID

All criteria were met	X
Criteria were not met and/or see below	

ACTION

### SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery. Matrix: solid/aqueous

SUPPOCATE COMPOUND

SAMPLEID	S1	S2	S3	S4	ACTION
_SURROGATE_ _LIMITS	STANDAR	DS_RECOVER	RIES_WITHII	N_LABORAT	ORY_CONTROL
S1 = o-Terpheny S3 = 1-Chlorooc				orobiphenyl omonaphthale	
QC Limits (%)* ( _LL_to_UL4 QC Limits* (Solid _LL_to_UL	40_to_140_	40_to_140_ to	_40_to_1	4040_to to	_140_

It is recommended that surrogate standard recoveries be monitored and documented on a continuing basis. At a minimum, when surrogate recovery from a sample, blank, or QC sample is less than 40% or more than 140%, check calculations to locate possible errors, check the fortifying standard solution for degradation, and check changes in instrument performance.

If the cause cannot be determined, reanalyze the sample unless one of the following exceptions applies:

- (1) Obvious interference is present on the chromatogram (e.g., unresolved complex mixture);
- (2) The surrogate exhibits high recovery and associated target analytes or hydrocarbon ranges are not detected in sample.

If a sample with a surrogate recovery outside of the acceptable range is not reanalyzed based on any of these aforementioned exceptions, this information must be noted on the data report form and discussed in the Executive Report. Analysis of the sample on dilution may diminish matrix-related surrogate recovery problems. This approach can be used as long as the reporting limits to evaluate applicable MCP standards can still be achieved with the dilution. If not, reanalysis without dilution must be performed.

All criteria were met _	_X
Criteria were not met and/or see below _	

## VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

MS/MSD Recoveries and Precision Criteria

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples.

At the request of the data user, and in consideration of sample matrices and data quality objectives, matrix spikes and matrix duplicates may be analyzed with every batch of 20 samples or less per matrix.

- Matrix duplicate Matrix duplicates are prepared by analyzing one sample in duplicate. The purpose of the matrix duplicates is to determine the homogeneity of the sample matrix as well as analytical precision. The RPD of detected results in the matrix duplicate samples must not exceed 50 when the results are greater than 5x the reporting limit.
- The desired spiking level is 50% of the highest calibration standard. However, the total concentration in the MS (including the MS and native concentration in the unspiked sample) should not exceed 75% of the highest calibration standard in order for a proper evaluation to be performed. The purpose of the matrix spike is to determine whether the sample matrix contributes bias to the analytical results. The corrected concentrations of each analyte within the matrix spiking solution must be within 40 140% of the true value. Lower recoveries of n-nonane are permissible but must be noted in the narrative if <30%.</p>

Sample ID:I	MC46129-1		Matrix	/Level:Soi	I
List the %Rs, R	PD of the compounds	which do no	t meet t	he QC criteria.	
MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
	3 872 -				
- 122					

Note: MS/MSD % recoveries and RPD within laboratory control limits. No MS/MSD analyzed for aqueous matrix in this data package. No action taken, blank spike/blank spike duplicate used to assess accuracy. % recoveries and RPD within laboratory control limits.

		C	criteria wer	All criteria w e not met and/or s	vere metX see below
No action is taken o informed professional conjunction with other data. In those instart affect only the samp However, it may be a systematic proble associated samples.	al judgment, the QC criteria ances where it the spiked, the letermined through	he data and deter can be o qualifica ough the l	reviewer r mine the i determined tion should MS/MSD re	may use the MS, need for some qual that the results the limited to thites esults that the lab	/MSD results in allification of the of the MS/MSD is sample alone. oratory is having
2. MS/MSD – U	nspiked Comp	ounds			
List the concentration compounds in the un					
COMPOUND	CONCENTRA SAMPLE	ATION MS	MSD	%RPD	ACTION
Criteria: None specifi	ed, use %RSD	) <u>&lt;</u> 50 as	profession	al judgment.	
Actions:					
If the % RSD > 50, q If the % RSD is not					

A separate worksheet should be used for each MS/MSD pair.

MSD, use professional judgment to qualify sample data.

			Criteria		Il criteria were me net and/or see be	
	VIII.	LABORATORY CON	ITROL SAMPL	E (LCS/LCS	SD) ANALYSIS	
matric		ata is generated to de	etermine accura	cy of the ar	nalytical method f	or various
	1.	LCS Recoveries Crit	eria			
		List the %R of compo	ounds which do	not meet t	he criteria	
LCS II	)	COMPOUND	% R	QC LIMIT	ACTION	
_LCS	S_RECO	OVERY_WITHIN_LAB	ORATORY_CO	ONTROL_L	IMTS	
		1.000 transport	931 18			<u> </u>
	Criteria * *	Refer to QAPP for sp The spike recovery r n-nonane are permis nonconformance in must be < 25%.	nust be betwee ssible. If the re	covery of n	-nonane is <30%	, note the
		s on LCS recovery see outside the %R and				
the as If the for the If more qualify	sociated %R of the affected that the affected that the second the	he analyte is > UL, que desamples and accept the analyte is < LL, que desamples analyte in the associal the compounds in the results as (J) an imples.	nondetects.  ualify all positive intensitive samples.  the LCS are no	e results (j)	and reject (R) n	ondetects
2.	Freque	ency Criteria:				
per ma If no, i the eff	atrix)? <u>Y</u> the data fect and	nnalyzed at the requirement of No.  a may be affected. Us a qualify data according the actions below	se professional	judgment to	o determine the	severity of

		Criteria were n	All criteria were metx_ ot met and/or see below	
IX.	FIELD	LABORATORY DUPLICATE PRECISION		
Sample	e IDs:	MC46109-3/MC46109-4	Matrix:Soil	

Field/laboratory duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which measures only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION	
Field duplicate analyzed with this data package. RPD within laboratory and generally acceptable control limits.						

### Criteria:

The project QAPP should be reviewed for project-specific information. RPD  $\pm$  30% for aqueous samples, RPD  $\pm$  50 % for solid samples if results are  $\geq$  SQL. If both samples and duplicate are  $\leq$ 5 SQL, the RPD criteria is doubled.

SQL = soil quantitation limit

#### Actions:

If both the sample and the duplicate results are nondetects (ND), the RPD is not calculable (NC). No action is needed.

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria.

If one sample result is not detected and the other is  $\geq 5x$  the SQL qualify (J/UJ).

**Note:** If SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is < 5x the SQL, use professional judgment to determine if qualification is appropriate.

All criteria were metX_	
Criteria were not met and/or see below	

#### XI. COMPOUND IDENTIFICATION

The compound identification evaluation is to verify that the laboratory correctly identified target analytes as well as tentatively identified compounds (TICs).

- 1. Verify that the target analytes were within the retention time windows.
  - Retention time windows must be re-established for each Target EPH
     Analyte each time a new GC column is installed, and must be verified and/or adjusted on a daily basis.
  - o The n-nonane (n-C9) peak must be adequately resolved from the solvent front of the chromatographic run.
  - o All surrogates must be adequately resolved from the Aliphatic Hydrocarbon and Aromatic Hydrocarbon standards.
  - For the purposes of this method, adequate resolution is assumed to be achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks.
  - The n-pentane (C5) and MtBE peaks must be adequately resolved from any solvent front that may be present on the FID and PID chromatograms, respectively.
- 1a. Aliphatic hydrocarbons range:
  - o Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for n-C9 and 0.01 minutes before the Rt for n-C19.
  - Determine the total area count for all peaks eluting 0.01 minutes before the Rt for n-C19 and 0.1 minutes after the Rt for n-C36.

Are the aliphatic hydrocarbons range properly determined?

Yes? or No?

Comments:

- 1b. Aromatic hydrocarbons range:
  - Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for naphthalene and 0.1 minutes after the Rt for benzo(g,h,i)perylene.
  - Determine the peak area count for the sample surrogate (OTP) and fractionation surrogate(s). Subtract these values from the collective area count value.

Are the aliphatic hydrocarbons range properly determined?

Yes? or No?

Comments:

		C	riteria were not		were metX_ see below
2.	If target analytes a laboratory resubmit t			identified,	request that th
3.	Breakthrough determevaluated for potentil % recovery of the frobasis by quantifying and aromatic fractionaphthalene or 2-m the total concentrator LCSD, fractionate	al breakthrough actionation surro naphthalene an ns of the LCS at the things and the things are the things and the things are	on a sample sp gate (2-bromor d 2-methylnaph and LCSD. If e ne in the alipha alene or 2-met	ecific basis naphthalene ithalene in lither the catic fraction hylnaphtha	by evaluating the and on a bate coth the aliphatic concentration of exceeds 5% of the LC
	NOTE:	methylnaphth summation	alene in the L0 of the conce ion and the co	CS/LCSD pontration de	thalene or 2 air includes th etected in th detected in th
	Comments:Conce_concentration_for_r				
4.	Fractionation Check Standard – A fractionation check solution is prepare containing 14 alkanes and 17 PAHs at a nominal concentration of 200 ng/µl each constituent. The Fractionation Check Solution must be used to evaluate the fractionation efficiency of each new lot of silica gel/cartridges, and establish the optimum hexane volume required to efficiently elute aliphatic hydrocarbons whi not allowing significant aromatic hydrocarbon breakthrough. For each analycontained in the fractionation check solution, excluding n-nonane, the Perce Recovery must be between 40 and 140%. A 30% Recovery is acceptable for nonane.				
	Is a fractionation che	ck standard ana	yzed?		Yes? or No?
	Comments: Not appl	icable.			

	All criteria were met _	_X
Criteria were	not met and/or see below	<i>!</i>

### XII. QUANTITATION LIMITS AND SAMPLE RESULTS

The sample quantitation evaluation is to verify laboratory quantitation results.

In order to demonstrate the absence of aliphatic mass discrimination, the response ratio of C28 to C20 must be at least 0.85. If <0.85, this nonconformance must be noted in the laboratory case narrative.

The chromatograms of Continuing Calibration Standards for aromatics must be reviewed to ensure that there are no obvious signs of mass discrimination.

Is aliphatic mass discrimination observed in the sample?

Yes? or No?

Is aromatic mass discrimination observed in the sample?

Yes? or No?

1. In the space below, please show a minimum of one sample calculation:

MC46109-5

EPH (C11 – C22, Aromatics)

RF = 98200

[] = (32347059)/(98200)

[] = 329.4 ppb Ok

MC46109-5

EPH (C9 - C18, Aliphatics)

RF = 73870

[] = (114572646)/(73870)

[] = 1,551 ppb Ok

- 2. If requested, verify that the results were above the laboratory method detection limit (MDLs).
- 3. If dilutions performed, were the SQLs elevated accordingly by the laboratory? List the affected samples and dilution factor in the table below.

SAMPLE ID	DILUTION FACTOR	REASON FOR DILUTION		
		<del>                                     </del>		
		<u></u>		
		1		
_		7		
		7.3		
		1		
	120 200			

If dilution was not performed, affected samples/compounds:	(J) for the affected	i compounds.	List the